Synthesis of Ionic Liquid Polymer Incorporating Activated Carbon for Carbon Dioxide (CO_2) Capture and Separation

by

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Dissertation submitted in partial fulfilment of

the requirements for the

Bachelor of Engineering (Hons)

(Chemical Engineering)

MAY 2013

Universiti Teknologi PETRONAS

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Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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Approved by,

_____

(Dr Muhammad Moniruzzaman)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

MAY 2013

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contain herein have not been undertaken or done by unspecified sources or persons.

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(Muhammad Hafiz 'Arif Bin Ahmad Sayukhi)

ABSTRACT

Carbon dioxide (CO_2) capture and separation by ionic liquids is one of the fastest growing branches in ionic liquids technology. Ionic liquids have emerged as potential solvents or materials for CO_2 capture and separation due to high chemical and thermal stability, virtually limitless chemical tunability, high ionic conductivity and their non-corrosive nature. The aim of this research project is therefore to synthesize ionic liquid polymer incorporating activated carbon and accompanying process for CO_2 capture and separation. The ionic liquid monomer of 1-Vinyl-3-ethylimidazalioum bromide, [Veim][Br] and 1-Vinyl-3-ethylimidazalioum bis(trifluoromethylsulfonyl)imide, [Veim][Tf_2N] were first synthesised to be used for the polymerization of the polymer material incorporating activated carbon. The purity of the [Veim][Br] and Veim][Tf_2N] has been verified using ^{1}H and ^{13}C NMR and also Ion Chromatography (IC) and both are proven to be in high purity. Following that, two ionic liquid based polymer materials, Poly(1-Vinyl-3-ethylimidazalioum bromide), Poly[Veim][Br] and also Poly(1-Vinyl-3-ethylimidazalioum bis(trifluoromethyl-sulfonyl)imide), Poly[Veim][Tf_2N] incorporating activated carbon were synthesized through free radical polymerization with 2,2'-azobis(isobutyronitirile) (AIBN) as the initiator with Trimethylolpropane tri-metacrylate (TRIM) as the crosslinker. The elemental composition of both polymer materials synthesized were examined using energy dispersive X-ray spectroscopy (EDX), in order to prove that activated carbon has been successfully incorporated into the polymer molecules. Furthermore, the morphology of both polymer materials has been investigated using field scanning emission scanning electron microscopy, (SEM). The results reveals that incorporating activated carbon leaded to the reduction in the number of large pore in the polymer materials demonstrating the role of activated carbon in the structure of the polymer material by uniformly covered on the polymer surface.

ACKNOWLEDGEMENTS

First and foremost, the author would like to express his highest gratitude to Allah the Almighty for making the Final Year Project (FYP) a successful and beneficial for him. In addition, it would not have been possible without the kind support and help from many individuals and organizations.

The author is highly indebted to Dr Muhammad Moniruzzaman for his guidance, advices, encouragement and constant supervision as well as providing necessary information in completing the project throughout the two semesters. Sincere appreciation goes to the coordinators of Final Year Project 1 and 2 for being supportive and cooperative throughout the project completion.

The gratitude is extended to all of the lecturers, technicians and technologists from Chemical Engineering department of Universiti Teknologi PETRONAS for their effort in helping and providing the author with all the necessary materials and other experimental tools required for the project.

Finally, the author's thanks and appreciations also go to his family and friends for their endless support and encouragement in making this project a success.

Thank you.

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CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

In the modern era of global industrialization, clean energy production has become one of the most predominant global issues. The reduction of greenhouse gases emission such as carbon dioxide configures prominently in the discussion on the future global energy policy.

Carbon dioxide (CO_2) is a colourless and odourless gas where it is naturally present in the atmosphere as part of the Earth carbon cycle which is the natural circulation of carbon among the atmosphere, oceans, soil, plant and animals. Besides, CO_2 is also well known as the primary greenhouses gas emitted from human activities such as combustion of fossil fuels for instances like coal, oil and natural gas for energy generation and transportation.

The combustion of fossil fuel to generate electricity has results in billions of tons annual worldwide CO_2 emission. According to the Figure 1.1, CO_2 emissions have accelerated in recent years where the worldwide growth rate in CO_2 emissions over the past decade is faster than that of previous decades. The graph shows that prior to 2002, the incremental annual increase had never reached 1 billion new metric tons of CO_2. Since 2002, 1 billion incremental tons have been added three times which were observed in the year of 2003, 2004, and 2010. In fact, 2010′s addition of 1.58 billion new tons globally is the largest annual increase on record [1].

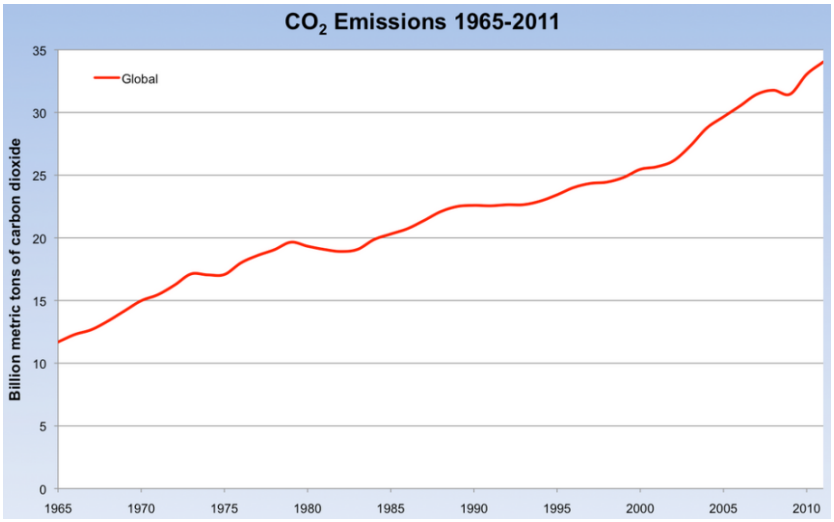


FIGURE 1.1. 2012 BP Statistical Review of World Energy [1]

Even with aggressive deployment of renewable energy resources like wind and solar, it is likely that it will be several decades before non-fossil fuel resources represent the majority of our electricity production [2]. Growing evidence of the link between anthropogenic CO_2 emissions and global climate change has therefore encouraged the development of a wide range of new CO_2 capture and separation technologies to curb CO_2 emissions. In addition, the development of CO_2 separation technologies can also be applied in the natural gas treating or sweetening process where undesirable contaminants such as hydrogen sulphide (H_2S) and CO_2 in the natural gas can be successfully removed.

Nowadays, there are many established technologies and ongoing research for CO_2 capture and separation such as physical and chemical absorption, adsorption, membranes and cryogenics. Figure 1.2 shows a wide range of technologies currently exist for CO_2 separation from gas stream although some of them have not been design for power-plant scale operations.

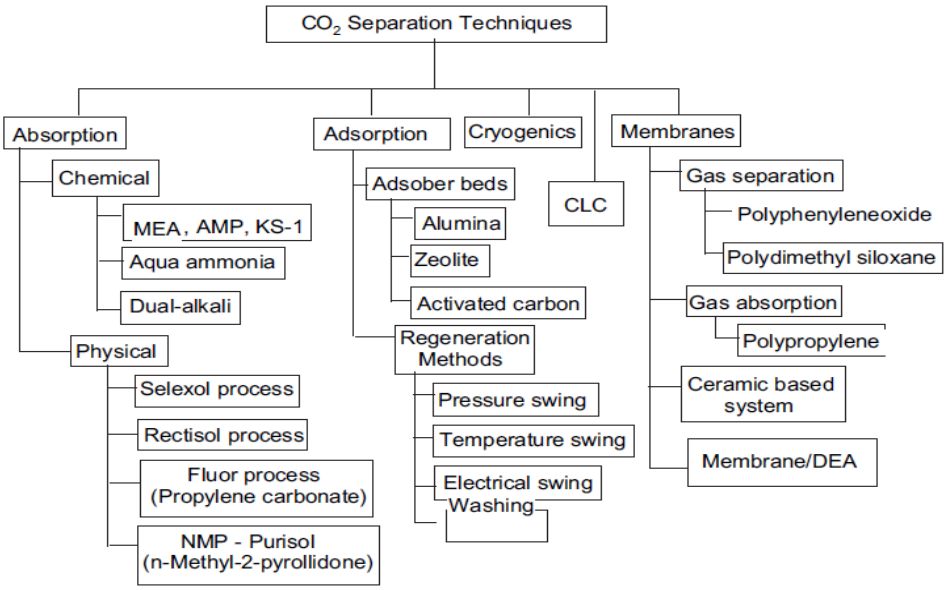


FIGURE 1.2. CO_2 Capture and Separation Techniques [3]

The absorption/stripping technology using amine solution such as monoethanolamine (MEA) is a commercialized technology used in natural gas industry for 60 years and is regarded as the most mature technology [3]. However, The MEA process suffers many disadvantages for CO_2 separation from flue gases such as low carbon dioxide loading capacity, high equipment corrosion rate, amine degradation by sulphur dioxide (SO_2), nitrogen dioxide (NO_2), hydrochloric acid (HCl) and hydrogen fluoride (HF) and oxygen in the flue gas which induces a high absorbent makeup rate and lastly high energy consumption during high temperature absorbent regeneration [3].

Therefore, developing the most economical and energy efficient CO_2 capture and separation technologies is urgently needed. The most attractive approach for the separation of targeted compound from a mixture of gases in a gas stream is selective absorption into a liquid [4]. A liquid that could facilitate the sequestration of gases without concurrent loss of the capture agent or solvent into the gas stream should prove to be a superior material in such applications [5]. To this end, ionic liquid have been proposed as solvents reagents for gas separations. Ionic liquids with a low melting point (less than 100°C) have been emerging as non-volatile and reversible absorbents for CO_2 capture. Compared to traditional organic solvents, the non-volatile ionic liquids are more environmentally friendly.

1.2 PROBLEM STATEMENT

Carbon dioxide (CO_2) capture and separations are crucial in the effort of the reduction of greenhouse gas emissions which contributes to climate change such as global warming. Besides, CO_2 separations are also vital in improving the value of natural gas through gas treating or sweetening. For this purpose, it is interesting to find out that ionic liquids have a great potential as an environmental-benign solvents due to their immeasurably low vapour pressure besides avoiding the lost of the sequestering agents during the CO_2 capture and separation process. Many studies have been done on the development of the ionic liquids for CO_2 separation since the first discovery of the solubility of CO_2 in ionic liquids. However, the absorption capacity of CO_2 in the ionic liquids can still be enhanced in many prospects because of their tuneable properties and synthetic structure which has make them opportunistic in CO_2 separation process. It is therefore proposed in this research to synthesize ionic liquid polymer incorporating activated carbon in order to increase the ionic liquid efficiency during CO_2 capture and separation.

1.3 OBJECTIVE

The objective of this research is to synthesize and characterize an ionic liquid based polymer incorporating activated carbon for CO_2 capture and separation. The polymer material formed is expected to have a significantly higher CO_2 absorption capacity compared to the conventional ionic liquids. This is because the polymer material formed should combine both absorption and adsorption mechanism of CO_2 into it in order to enhance the CO_2 solubility.

1.4 SCOPE OF STUDY

In order to achieve the objective of this research project, the scope of study for this research will cover:

1. Development and synthesis of ionic liquid based polymer incorporating activated carbon. Ionic liquid monomers, [Veim][Br] and [Veim][TF_2N] is going to be synthesized and activated carbon is going to be microencapsulated in surfactant aggregates formed in the ionic liquid monomers which will then be incorporated into polymer frameworks through polymerization.
2. The study the characteristics of the polymer material formed. The polymer material formed will be investigated for their external morphology (texture), chemical composition, crystalline structure and orientation of materials making up the polymer material.

1.5 RELEVANCY AND FEASIBILITY OF THE PROJECT WITHIN THE SCOPE AND TIME FRAME

The synthesis of ionic liquid polymer incorporating activated carbon for CO_2 capture and separation is significant as the removal of CO_2 from the flue gas of existing power generation and natural gas is likely to be an important component of our energy portfolio in this decades. Ionic Liquids have been well known as an energy efficient liquid absorbent for CO_2 capture and therefore the incorporation of activated carbon into it is expected to have many new advantages to be applied in the industry.

The author was given 28 weeks effective from 14th January 2013 to conduct the research project under the supervision of Dr Muhammad Moniruzzaman from Chemical Engineering Department, Universiti Teknologi PETRONAS. An organized and suitable Gantt chart has been established to ensure that the project is feasible within the timeframe of 7 months and also under the scope that has been set up. All of the raw materials, chemical instruments and facilities are provided in the Universiti Teknologi PETRONAS so that the project is feasible to be conducted. Regular meeting and supervision from the supervisor while conducting the project helped the author a lot in achieving the objective of this project.

CHAPTER 2

LITERATURE REVIEW

This chapter will review and discuss on the relevant results with regards to CO_2 capture and separation by ionic liquids. It comprises of some general description on characteristics of ionic liquids, CO_2 separation by room temperature ionic liquids (RTILs) and task specific ionic liquids (TSILs), activated carbon for CO_2 adsorption and dispersion of carbon nanotubes in ionic liquids.

2.1 GENERAL CHARACTERISTICS OF IONIC LIQUIDS

Ionic Liquids (ILs) are usually composed of large asymmetric organic cations and inorganic or organic anions. They exhibit unique properties compared to other solvents. ILs are generally colourless liquids with relatively high viscosities. Besides, they exhibit very low vapour pressures under ambient conditions and thus are effectively non-volatile. In addition, ILs are good solvents for a broad spectrum of inorganic, organic and polymeric materials and are immiscible with numerous organic solvents. Some properties, such as the thermal stability and miscibility, mainly depend on the anion, while others, such as the viscosity, surface tension and density, depend on the length of the alkyl chain in the cation and/or shape or symmetry. [6] Some typical cations and anions constituting ILs are shown in Figure 2.1 and Figure 2.2.

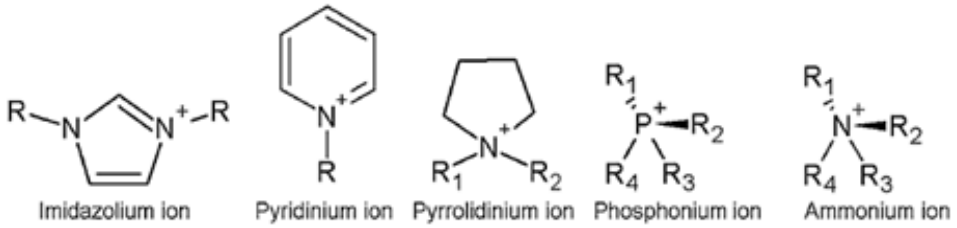


FIGURE 2.1. Typical Cations Constituting Ionic Liquids [7]

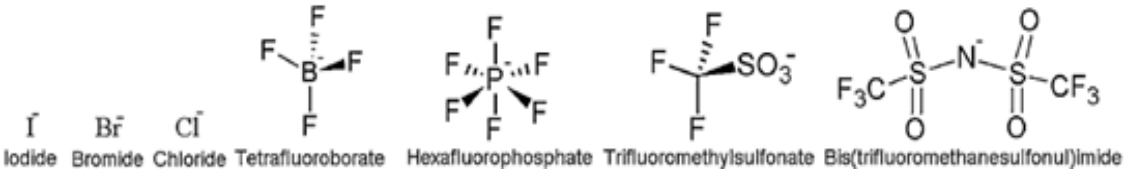
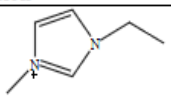
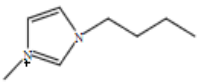
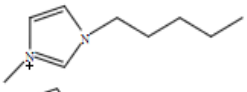
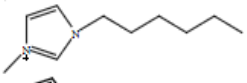
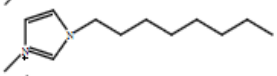
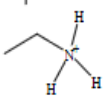
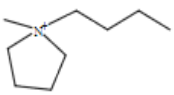


FIGURE 2.2: Typical Anions Constituting Ionic Liquids [7]

The most commonly employed ILs anions are polyatomic inorganic species, such as [PF_6^-] and [BF_4^-]. The most prominent cations are pyridinium and an imidazolium ring species with one or more alkyl groups attached to the nitrogen or carbon atoms. The physical properties of some common ionic liquids are presented in Table 2.1.

TABLE 2.1. Physical Properties of Ionic Liquids in Separation Technology [6]

Chemical formula		Abbreviation	Melting point, °C	Density (g mL^{-1}), 25 °C	Viscosity (cP), 25 °C	Molecular weight
Cation	Anion					
	$[BF_4]^-$	[EMIM][BF_4]	6	1.248	66	197.8
	$[PF_6]^-$	[EMIM][PF_6]	58-62	1.373	450	256.13
	$[BF_4]^-$	[BMIM][BF_4]	-82	1.208	233	225.80
	$[PF_6]^-$	[BMIM][PF_6]	10	1.373	400	284.18
	$[Br]^-$	[BMIM]Br	60	1.134	Solid	218.9
	$[Cl]^-$	[BMIM]Cl	89	1.120	Solid	146.50
	$[CF_3SO_3]^-$	[BMIM][CF_3SO_3]	16	1.290	90	260.0
	$[(CF_3SO_2)_2N]^-$	[BMIM] [$(CF_3SO_2)_2N$]	-4	1.420	52	487.9
	$[NTfO_2]^-$	[BMIM] [$NTfO_2$]	-8	1.404	48	433.0
	$[BF_4]^-$	[AMIM][BF_4]	-88	1.231	321	240.02
	$[BF_4]^-$	[HMIM][BF_4]	-82	1.075	211	254.08
	$[PF_6]^-$	[HMIM][PF_6]	-61	1.304	800	312.00
	$[BF_4]^-$	[OMIM][BF_4]	-79	1.11	440	281.8
	$[Cl]^-$	[OMIM][Cl]	0	1.000	16,000	230.50
	$[NTfO_2]^-$	[MPPyr] [$NTfO_2$]$^-$	0	1.44	39	416
	$[HCOO]^-$	BAF	-10	0.99	11.5	91
	$[NTfO_2]^-$	[BMPyrrol] [$NTfO_2$]	-50	1.4	71	422

In addition to the interactions that exist in conventional organic solvents (hydrogen bonding, dipole–dipole and van der Waals interactions), ILs also have ionic interactions which are the mutual electrostatic attraction or repulsion of charged particles, making them very miscible with polar substances. Hydrogen bonding is

thought to exist between the oxygen or halide atoms on the anion and the hydrogen atoms on the imidazolium or pyridinium ring of the cation in ILs. At the same time, the presence of the alkyl chain on the cation determines the solubility of the ILs in less polar fluids. Furthermore, the properties of the ILs can significantly change by varying the length of the alkyl groups that are incorporated into the cation and the types of anions. For example, for the 1-alkyl-3-alkylimadazolium cation, replacing the [PF_6^-] anion with [BF_4^-] dramatically increases the solubility of the IL in water, whereas replacing the anion with the [Tf_2N^-] ion decreases the water solubility. The change in the length of the 1-alkyl chain from 1 to 9 on 1-alkyl-3-methylimadazolium hexafluorophosphate [$C_n$mim][PF_6] can make the usually soluble ILs very immiscible in water. [6]

2.2 CO_2 SEPARATION BY ROOM-TEMPERATURE IONIC LIQUIDS (RTILS)

Blanchard *et al*. firstly reported that CO_2 is highly soluble in ionic liquids of 1-butyl-3-methylimidazolium hexafluorophosphate ([C_4mim][PF_6]), reaching a mole fraction of 0.6 at 8 MPa, while at 13.8 MPa and 40 °C the solubility of [C_4mim][PF_6] in CO_2 is less than 10^{-5} mole fraction [8].

The solubility of CO_2 in a series of imidazolium based ILs at low pressure has been determined by Baltus *et al*. The solubility of CO_2 was found to increase with the length of the alkyl side chain on the imidazolium ring but the CO_2 solubility in ILs with phenyl groups was lower when compared to that with alkyl groups. Furthermore, the CO_2 solubility is greater in ionic liquids with [Tf_2N^-] anions than that in ILs with [PF_6^-] anions. Besides, the imidazolium-based ionic liquids with a fluorine-substituted C_8 side chain are higher than the corresponding ILs shaving a non-fluorinated C_8 side chain [9].

In addition, Anderson *et al*. studied the impact of fluorination of the cation on CO_2 solubility with the conclusion that the CO_2 solubility was higher in [$C_8H_4F_{13}$mim][Tf_2N] than [$C_6H_4F_9$mim][Tf_2N], and lowest in [C_6mim][Tf_2N]. In all, the CO_2 solubility increases as the numbers of fluorine in the alkyl side chain but this tendency is not very visible [10].

Past research have shown that some conventional room-temperature ionic liquids especially imidazolium based, have diminished corrosion of the equipment, and the heat capacity of RTIL is almost one-third of that of aqueous systems, which may have profound effect in reducing the high investment and operation cost. As for the regeneration of the RTIL based materials, it has be found that it can be easily recycled either by pressure sweep process coupled with vacuum treatment, or by applying heat or by bubbling nitrogen through the absorbent [8].

Although many studies have shown that the conventional RTILs can absorb and separate CO_2 effectively to a certain extent, it is just by mechanism of physical adsorption. The drawback is that the CO_2 absorption capacity of these ILs is far below that of the traditional alkanolamine solutions even in the fluorinated ILs.

2.3 CO_2 SEPARATION BY TASK SPECIFIC IONIC LIQUIDS (TSILS)

Past studies as have been discussed earlier have give indications that CO_2 is sufficiently soluble in room-temperature ionic liquids (RTILs). However, the CO_2 capture ability can be improved by introducing basic character in the ILs.

Davis *et. al.* reported that functionalization of ionic liquids with a suitable moiety (like amine) may be implied to enhance the CO_2 capture ability of ILs. CO_2 absorption ability of Task Specific Ionic Liquids (TSILs) can reach up to threefold of the corresponding RTILs. Besides, Sanchez *et. al.* has studied that the enhanced effect of pressure in case of TSILs was observed by the fact that there was a steady increase in gas load with rise in pressure, providing evidence both for chemical as well as physical sorption. The effect is not so apparent in case of aqueous amine solutions which possess stoichiometric limitations [11].

In fact, it has been understood that reversible sequestration of CO_2 is achieved by attaching primary amine moiety to an imidazolium cation, without any reduction in the ionic-liquid stability. Davis *et. al.* has discovered that for five consecutive cycles of gas absorption, the regenerated TSIL 1-n-propylamine-3-butylimidazolium tetrafluoroborate [pabim][BF_4] did not show any loss of efficiency. The TSIL when exposed to CO_2 for 3 hours at room temperature and pressure, the mass gain was 7.4% which corresponds to 0.5 molar uptake of CO_2 which has reached the maximum theoretical value for CO_2 capture as amine carbamate [5].

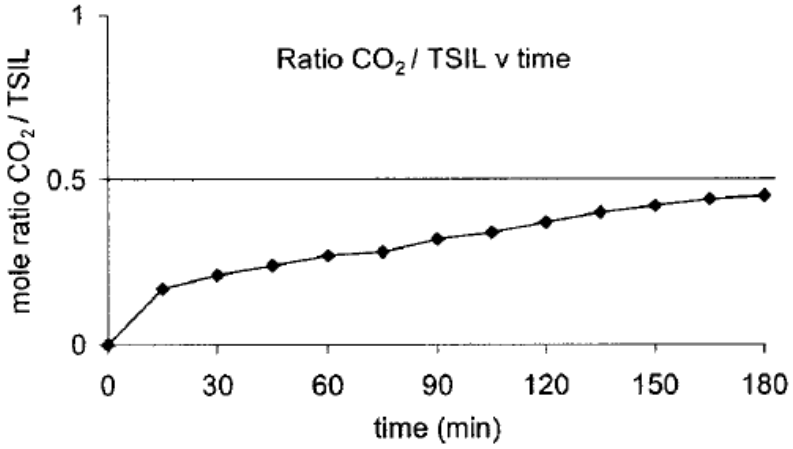


FIGURE 2.3. CO_2/TSIL Molar Ratio as a Function of Time [5]

In addition, the TSIL remains stable even after five gas sorption/desorption cycles without any detectable loss in efficiency. The inclusion of water in the ionic liquid was found to increase the CO_2 holding capacity which might be due to the formation of bicarbonate species as well [5]. The proposed mechanism of interaction between CO_2 and [pabim][BF4] according to Davis *et. al* is shown in Figure 2.4.

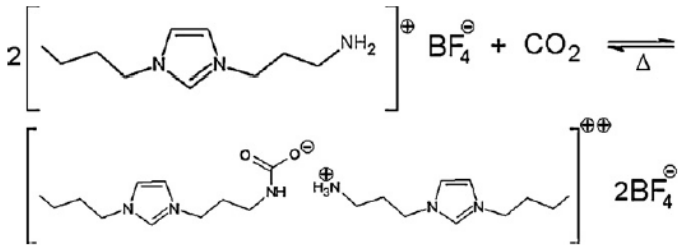


FIGURE 2.4. Proposed Mechanism for CO_2 Capture by [pabim][BF4] [5]

CO_2 absorption ability increases significantly on functionalization of ionic liquid with primary amine moiety. According to Rahman *et. al*., task-specific ionic liquids 1-allyl-3-methylimidazoluim tetrafluoroborate [Amim][BF_4] and 1-allyl-3-methylimidazoluim dicyanamide [Amim][DCA], perform like chemical solvents at low pressures which is at around 1 bar, however, at higher pressures they pursued the performance of room-temperature ionic liquid, 1-butyl-3-methylimidazoluim tetrafluoroborate [bmim][BF_4]. On the other hand, aqueous amine solutions accomplished the maximum capacity at about 2 bar and any further increase in pressure does not seem feasible. Whereas functionalized ionic liquids (TSILs) carry on steady CO_2 absorption with ascending pressure [11]. This behaviour represents that TSILs possess both chemical as well as physical tools for CO_2 separation.

However, Gutowski *et. al.* found that CO_2 capture results in sharp increase of viscosity of the TSILs, resulting into a gel-like material. This drawback maybe avoided by utilizing mixtures of TSILs and RTILs or TSILs may be adsorbed onto porous membranes [12]. Thus in spite of the tunable approach towards TSILs, these functionalized species exhibit much higher viscosities as compared to the corresponding RTILs or other commercially available CO_2 scrubbing solutions. This high viscosity might pose serious complications to be applicable on industrial scale of CO_2 separation process.

2.4 ACTIVATED CARBON FOR CO_2 ADSORPTION

Activated carbon is the collective name for a group of porous carbons manufactured by the treatment of a char with oxidizing gases or by carbonization of carbonaceous materials impregnated with dehydrating chemicals [13]. All these carbons exhibit a high degree of porosity and an extended internal surface area.

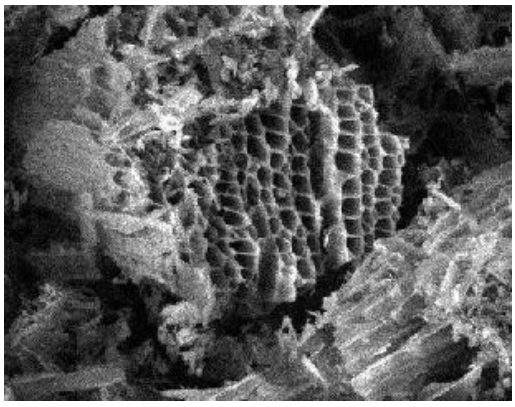


FIGURE 2.5. Activated Carbon as Viewed by an Electron Microscope [14]

The main application of activated carbon is found in the removal of impurities from gases and liquids by an adsorption process. There are many studies that have shown activated carbon can be used for CO_2 capture and separation. The surface of activated carbon can bind molecules from the liquid or gas phases by predominantly physical forces of the van der Waals type, causing a higher concentration of the adsorbate at the interface than in the bulk fluid. However, there is also the possibility of chemisorption, caused by stronger valence forces on the so-called active sites of the carbon surface. The efficiency of activated carbon will, therefore, depend on its accessible surface area where physical adsorption takes place, and also on the presence of active sites at which chemisorption may occur [13]. Due to the good adsorption ability of the activated carbon towards CO_2, it is proposed that the

activated carbon is dispersed and polymerized in the ionic liquid in order to enhance the ionic liquid ability for CO_2 capture and separation.

2.5 DISPERSION OF CARBON NANOTUBES IN IONIC LIQUIDS

By far, there is no studies that has been done on the dispersion and incorporation of activated carbon in ionic liquids. Therefore it is important to study the characterization and potential application of the carbon nanotubes-ionic liquids (CNTs-ILs) hybrids in order to understand the dispersion of activated carbon in ionic liquids. The number of scientific papers published each year on the dispersion of the CNTs in ILs has been steadily increasing since combination of ILs and CNTs was firstly reported in 2003, when Fukushima *et. al.* found that CNTs could form gelatinous materials, also called bucky gels upon being ground with room temperature ILs (RTILs) [23]. CNTs are unique form of carbon consists of single cylindrical wall CNTs (SWCNTs) and multiple concentric cylinders CNTs (MWCNTs) [15].

In general, it is found that CNTs in the forms of SWCNTs and MWCNTS can be easily dispersed in the imidazolium based RTILs. In many research for the preparations of CNTs-IL hybrid where ILs have been confined in CNTs, ILs are coated on the outer surface of CNTs via covalent or non-covalent methods. While the ILs can be used in small quantities to disperse CNTs in a matrix solution, they can as well act as a binder between CNTs and another component. ILs can also be the main component of the hybrid in which CNTs can disperse readily without any additional dispersing agent. [15]

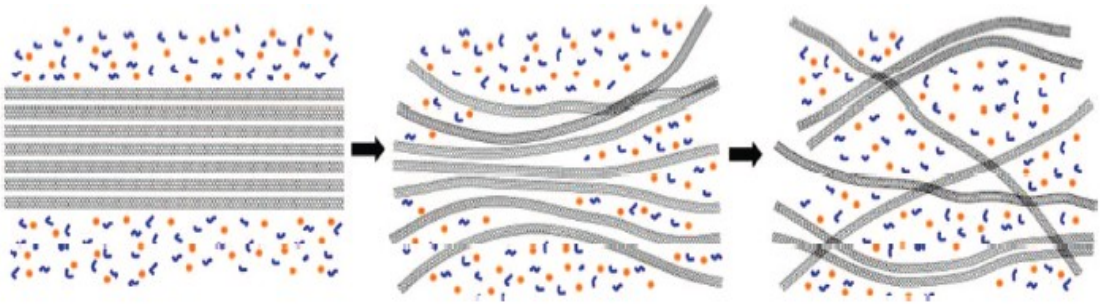


FIGURE 2.6. Schematic Diagram of Dispersion Process for CWNTs in Ionic Liquids [16].

Most of the researchers have assumed that the ILs interact with CNTs through cation-π and/or π- π interactions [15]. However, Wang and co-workers carried out a systematic study where the Raman and IR measurements on the mixtures of ionic

liquids and SWCNTs as well as other materials all unambiguously show no such strong interaction such as cation-π interaction exist between SWCNTs and imidazolium ions. The overall organization of is not remarkably altered by SWCNTs in which however, the shielding effects of the ionic liquid on the π- π stacking interaction among SWCNTs takes the key role in dispersing the SWCNTs. The ionic liquid is therefore interact with SWCNTs through weak van der Waals interactions [16]. The ability of the ILs to disperse the CNTs has therefore provided some useful insights for the dispersion of the activated carbon in the ILs that will be conducted in this research project for polymerization purpose.

CHAPTER 3

METHODOLOGY

3.1 RESEARCH METHODOLOGY

The methodology for conducting this research project will be based on exploration and discovery. This methodology can be achieved through laboratory experiments approach which has been divided into three steps which include preliminary works, experimental works, discussions and conclusions.

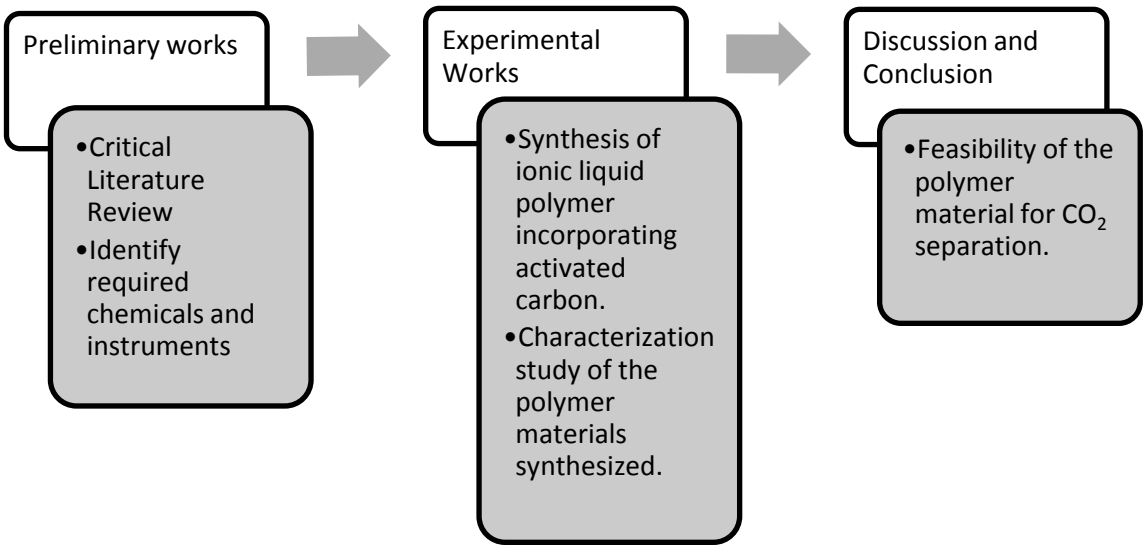


FIGURE 3.1. Schematic Diagram of Research Methodology

In the preliminary works, the area and scope of this research and literature review is narrowed down to the synthesize of ionic liquid polymer incorporating activated carbon for CO_2 separation so that it is feasible and could be completed within the allocated time frame. After a critical literature review has been done, the laboratory experiments can be conducted provided the required chemicals and equipments are available in order to achieve the objective of this research. Experimental works will

start with synthesis of the polymer material and then followed by the characterization study of the polymer materials synthesized. Finally, the characteristic of the polymer material formed is going to be investigated and the feasibility of CO_2 in the polymer material will be evaluated in order to see if the objective of this research project is achieved.

3.2 PROJECT ACTIVITIES

3.2.1 Requisition of Materials

Chemicals that will be used in the experimental works are being listed as following:

1. 1-Vinyl Imidazole, $C_5H_6N_2$.
2. Bromoethane, C_2H_5Br.
3. Ethyl Acetate, $C_4H_8N_2$.
4. Acetonitrile, C_2H_3N.
5. Ethanol, C_2H_5OH.
6. Lithium bis(Trifluoromethyl-sulfonyl) imide, $C_2F_6LiNO_4S_2$.
7. Trimethylolpropane tri-metacrylate, $C_{18}H_{26}O_6$ for crosslinker.
8. 2,2'-azobis(isobutyronitirile), $C_8H_{12}N_4$ for initiator.
9. Activated Carbon.

3.2.2 Requisition of Equipments

Tools that will be used for the experiment works are being listed as following:

1. Round Bottom Flux
2. Reflux system
3. Stirrer
4. Oil/Water Bath
5. Hot plate

In addition, equipments that will be used through out the experiment works are being listed as following:

1. Freeze-dryer
2. Scanning Electron Microscope (SEM)

3.2.3 Experimental Design / Procedure

The experimental procedures of this research are planned to be conducted with the raw materials, chemical instruments and facilities provided in the Universiti Teknologi PETRONAS. The experimental procedures of this research is basically divided into five main steps shown as following:

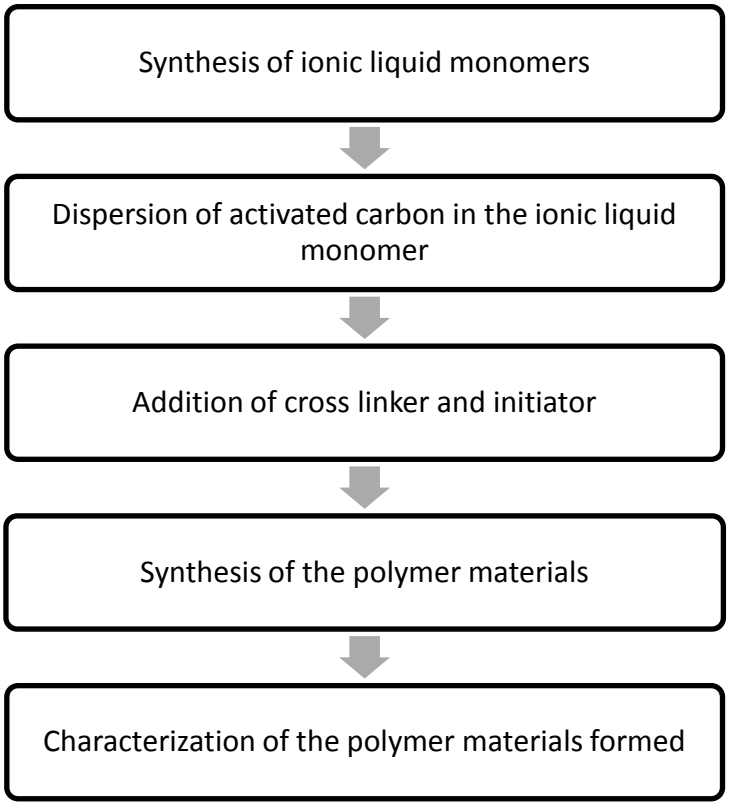


FIGURE 3.2. Schematic Diagram of The Experimental Procedures

3.2.3.1 Synthesis of Ionic Liquid Monomer

Ionic Liquid monomer 1-Vinyl-3-ethylimidazolium (Trifluoromethylsulfonyl)imide ([Veim][TF_2N]) is going to be synthesized through an anion exchange between 1-Vinyl-3-ethylimidazolium bromide ([Veim][Br]) and Lithium bis(Trifluoromethyl-sulfonyl)imide (Li[TF_2N]). In the preparation of [Veim][Br], Bromoethane is going to be added dropwise to 1-Vinylimidazole with vigorous stirring in an ice-bath, following by heating in oil bath for a few hours. The resulting white solid, [Veim][Br] is going to be washed with Ethyl Acetate several times to remove unreacted materials then followed by rotary evaporator followed by freeze-drying for 24hours. Following that, anion exchange is going to be conducted in molar ratio 1:1 between Li[TF_2N] and [Veim][Br] to obtain the ionic liquid monomer

[Veim][TF_2N]. The product is stored in a desiccator to avoid water absorption due to its hygroscopicity.

3.2.3.2 Incorporation of activated carbon into IL polymer material

Activated carbon is going to be microencapsulated in surfactant aggregates formed in ionic liquid monomer or the solution of ionic liquid monomer and then incorporated into polymer frameworks through polymerization of [Veim][Br] and [Veim][TF_2N]. Trimethylolpropane tri-metacrylate, TRIM is going to be used as crosslinker and the polymerization of [Veim][TF_2N] is going to be started by adding initiator 2,2'-azobis(isobutyronitirile), AIBN (3-5 mol% based on ionic liquid monomer). After a few hours later, the polymer material formed is going to be washed a few times with to remove unreacted substances and precipitate the polymer materials.

3.2.3.3 Characterization of the Polymer Material formed

After the fabrication process of the polymer material is done, the characterization of the polymer material is going to be investigated using scanning electron microscope (SEM). The signals that derive from electron-ionic liquid polymer material interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the polymer materials.

3.3 KEY MILESTONES

In order to meet the objective of this research, several key milestones have been identified and set up as following:

1. Identify the purpose of this research project.
2. Literature review to gather information from variety of sources such as journals and books on ionic liquids, activated carbon adsorption principle, and dispersion of activated carbon in ionic liquids .
3. Design the experimental procedures to synthesize ionic liquid polymer incorporating activated carbon.
4. Data analysis and interpretation of the results obtained.
5. Proper documentation and reporting of the findings of this research project.

3.4. STUDY PLAN (GANTT CHART)

The study plan for the research project has been set up and shown in the table as following:

TABLE 3.1. Research Project Gantt Chart

Activities	FYP 1														FYP 2													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Selection of Project Title																												
Critical Literature Review on Ionic Liquids, Activated Carbon, And Dispersion Of Activated Carbon In Ionic Liquids																												
Requisition of Chemicals And Equipments																												
Synthesis of Ionic Liquid Polymer Incorporating Activated Carbon																												
Characterization of The Polymer Material Formed																												
Data Analysis And Interpretation																												

CHAPTER 4

RESULTS AND DISCUSSION

4.1 SYNTHESIS AND PREPARATION OF IONIC LIQUID MONOMER 1-VINYL-3 ETHYLIMIDAZALIOUM BROMIDE

1-Vinyl-3-ethylimidazalioum bromide ($C_7H_{11}N_2Br$) or also known as [Veim][Br] is prepared by one step quaternization reaction of 1-Vinyl imidazole with a halo-alkane compound where the positively charged imidazolium ring is directly connected to a vynil group (Figure 4.1).

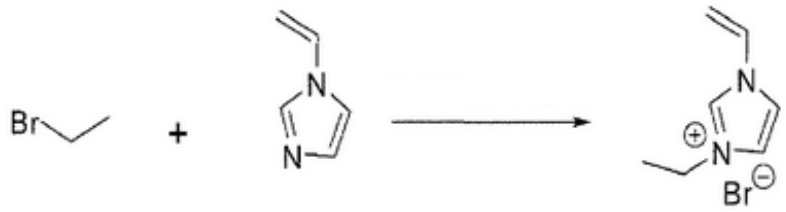


FIGURE 4.1. Synthesis route of 1-Vinyl-3-ethylimidazalioum bromide

The 1-Vinyl-3-ethylimidazalioum bromide was synthesized in the reaction of 1-Vinylimidazole with bromoethane. Bromoethane (33g, 0.3mol) was added dropwise to 1-Vinylimidazole (28g, 0.3mol) with vigorous stirring in ice bath followed by heating in oil bath at 40°C for 2 hours. The resulting white solid was washed with ethyl acetate several times to remove unreacted materials then dried using rotary evaporator, followed by freeze drying for 24 hours and left in a vacuum oven for 2 days. The synthesis steps are being shown in Figure 4.2.

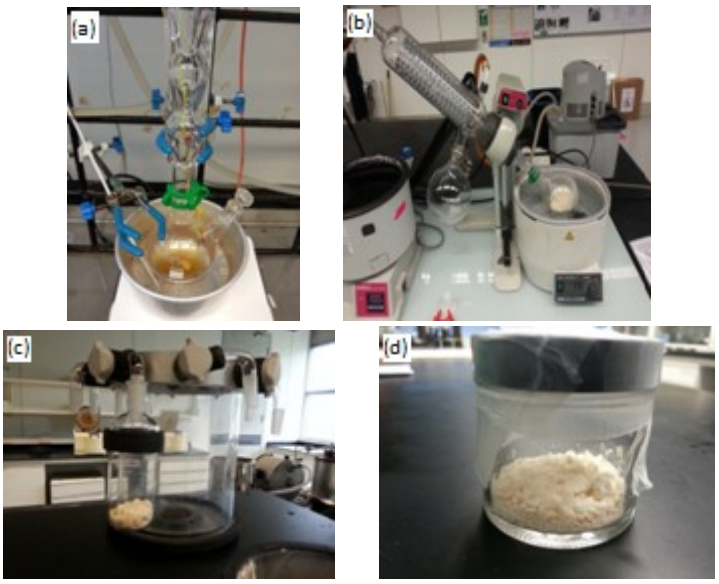


FIGURE 4.2. Synthesis steps of [Veim][Br] **(a)**Reaction at 40°C for 2 hours **(b)**Dried using rotary evaporator after washing with ethyl acetate **(c)**Further dried in freezedryer **(d)**Resulting white solid of [Veim][Br]

The purity of this [Veim][Br] was verified by ^{1}H NMR. and ^{13}C NMR. **^{1}H NMR** in Figure 4.3 (500MHz; MeOD, δ/ppm): 9.50(s,1H), 8.20(s,1H), 7.65(s,1H), 7.30(dd,1H), 5.90(dd,1H), 5.42(dd,1H), 4.20(q,2H), 1.45(t,3H). **^{13}C NMR** in Figure 4.4 (500MHz; MeOD, δ ppm): 15.50, 49.05, 110.00, 120.80, 124, 130.00, 136.00.

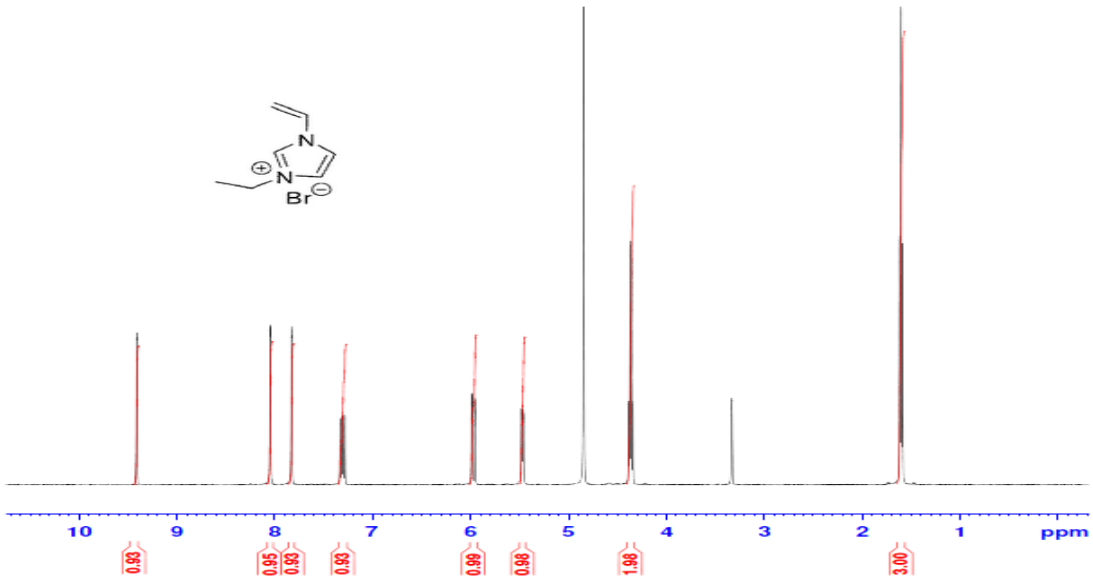


FIGURE 4.3. 500 MHz ^{1}H NMR spectra of [Veim][Br]

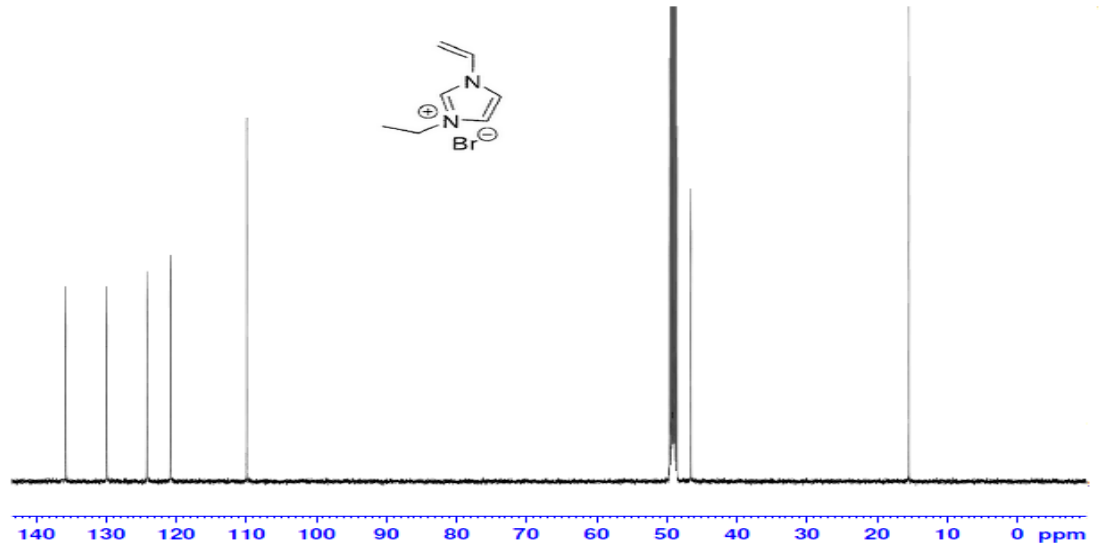


FIGURE 4.4. 500 MHz ^{13}C NMR spectra of [Veim][Br]

4.2 SYNTHESIS AND PREPARATION OF IONIC LIQUID MONOMER 1-VINYL-3-ETHYLIMIDAZALIOUM BIS(TRIFLUOROMETHYL-SULFONYL)IMIDE

1-Vinyl-3-ethylimidazalioum bis(trifluoromethylsulfonyl)imide ($C_9H_{11}F_6N_3O_4S_2$) or also known as [Veim][Tf_2N] is a viscous, yellowish ionic liquid and is prepared via anion exchange between the ionic liquid monomer 1-Vinyl-3-ethylimidazalioum bromide [Veim][Br] with Lithium bis(trifluoromethylsulfonyl)imide Li[TF_2N] in a molar ratio of 1:1.1 to produce [Veim][Tf_2N] (Figure 4.5).

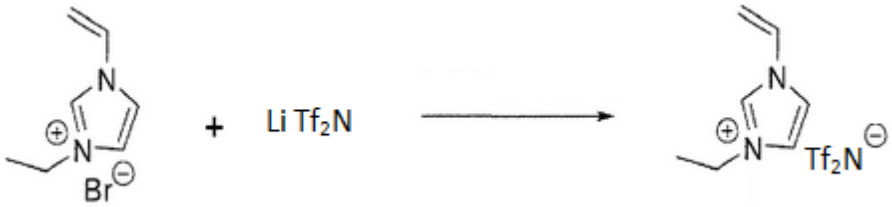


FIGURE 4.5. Synthesis route of 1-Vinyl-3-ethylimidazalioum bis(trifluoromethylsulfonylimide).

Ionic liquid monomer [Veim][Br] (12.788g, 0.063mol) was mixed with 6mL of distilled water. Li[Tf_2N] (20g, 0.064mol) was then added drop wise to minimise the amount of heat generated and mix with constant stirring at room temperature until the formation of two phase. The bottom phase and the top phase were contained [Veim][Tf_2N] and aqueous Li[Br] respectively. After decanting the top phase, the ionic liquid was washed thoroughly with 5mL of distilled water three times until the washing is totally Br^- free. The distilled water used for washing is then removed with

vacuum liner followed by lyophilization in vacuum oven. The synthesis steps are being shown in Figure 4.6.

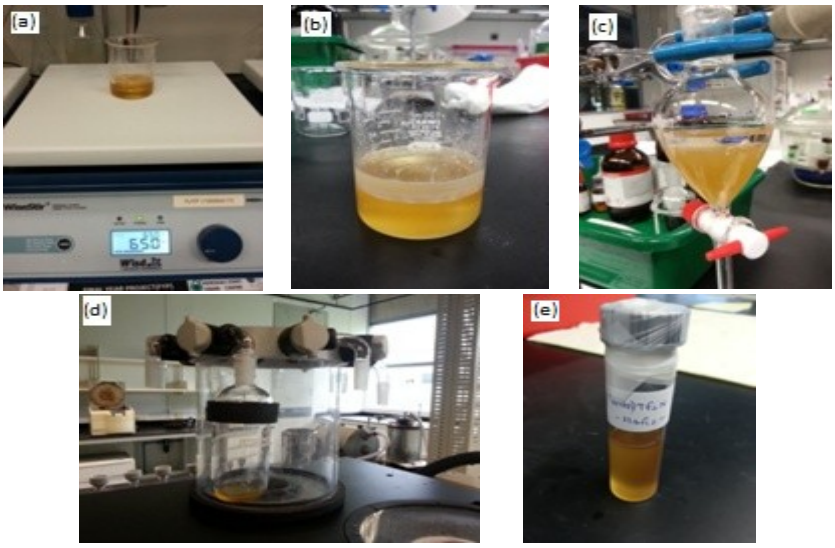


FIGURE 4.6. Synthesis steps of [Veim][Tf_2N] **(a)**Reaction at room temperature for 1 hour **(b)**Decanting the top phase of LiBr from the [Veim][Tf_2N] **(c)**[Veim][Tf_2N] after washing with distilled water **(d)** Dried in freezedryer after washing **(e)**Resulting yellowish viscous liquid of [Veim][Tf_2N]

The purity of this [Veim][Tf_2N] was verified by ^{1}H NMR. and ^{13}C NMR. **^{1}H NMR** in Figure 4.7 (500MHz; MeOD, δ ppm): 9.20(s,1H), 7.95(s,1H), 7.75(s,1H), 7.25(dd,1H), 5.90(dd,1H), 5.45(dd,1H), 4.30(q,2H), 1.60(t,3H). **^{13}C NMR** in Figure 4.8 (500MHz; MeOD, δ ppm): 15.00, 49.00, 110.00, 119.5, 121.00, 122.05, 124.00, 129.50, 146.05

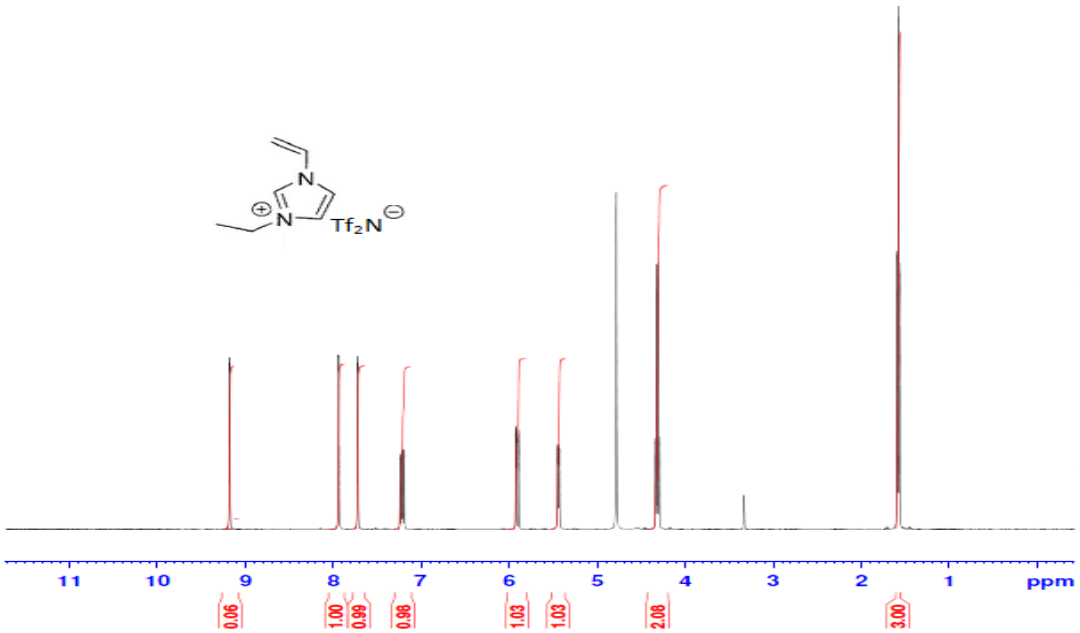


FIGURE 4.7. 500 MHz ^{1}H NMR spectra of [Veim][Tf_2N]

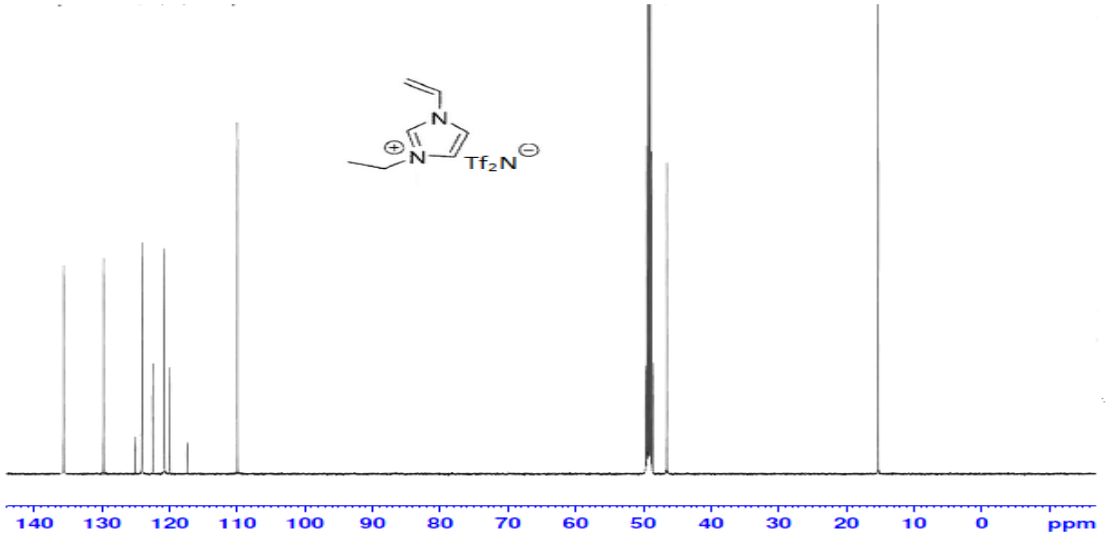


FIGURE 4.8. 500 MHz ^{13}C NMR spectra of [Veim][Tf_2N]

In addition, the purity of this [Veim][Tf_2N] was further verified by ion chromatograph (IC) to measure the traces of Br^- left in the [Veim][Tf_2N] after the anion exchange. From Figure 4.9 The actual chromatogram of Br^- in the [Veim][Tf_2N] was calculated to be 2628.86 ppm, after the chromatogram of Br^- in Figure 18 which is 18.929 ppm of Br^- has been multiplied with the dilution factor used in the ion chromatograph. The concentration of Br^- in the [Veim][Tf_2N] is in the acceptable amount so the[Veim][Tf_2N] produced is still considered as in high purity.

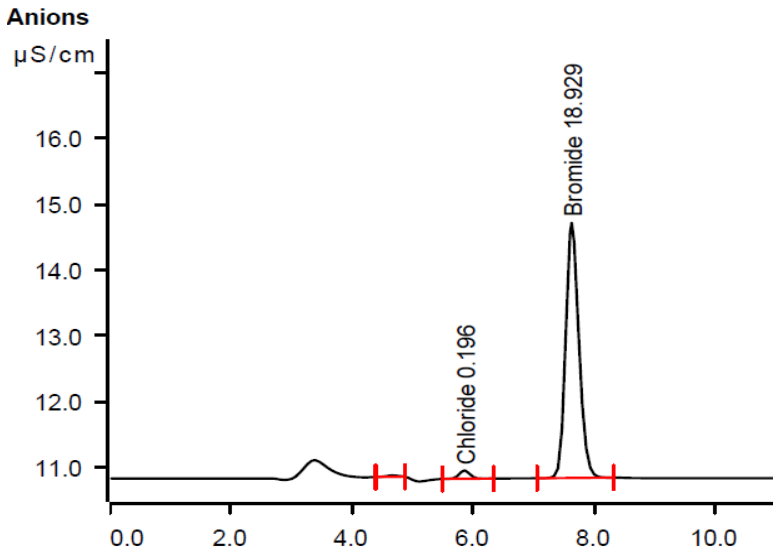


FIGURE 4.9. Ion Chromatograph of Br^- in [Veim][Tf_2N]

4.3 SYNTHESIS OF IONIC LIQUID POLYMERS POLY[VEIM][BR] AND POLY[VEIM][BR] INCORPORATING ACTIVATED CARBON

Poly(1-Vinyl-3-ethylimidazalioum bromide), the Poly[Veim][Br] was synthesized by conventional free-radical polymerization. The polymerization of ionic liquid monomer [Veim][Br] was initiated by addition of 2,2'-azobis(isobutyronitirile) (AIBN) together with Trimethylolpropane tri-metacrylate (TRIM) as the crosslinker. The synthesis route is illustrated as in Figure 4.10.

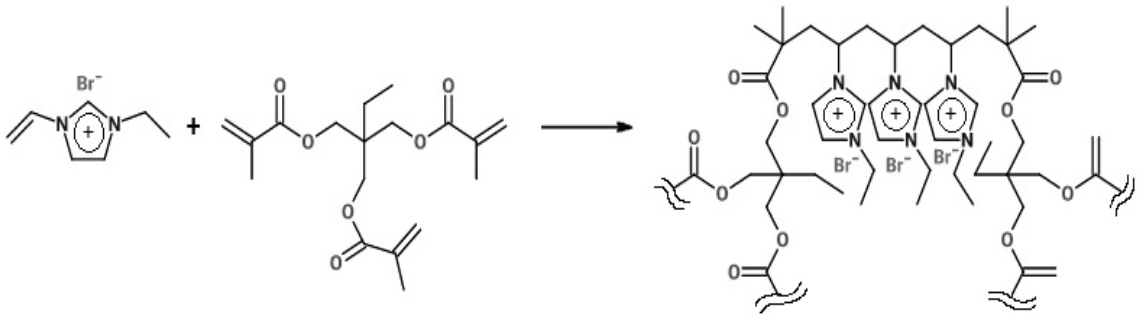


FIGURE 4.10. Synthesis Route of Poly(1-Vinyl-3-ethylimidazalioum bromide)

In the synthesis of the Poly[Veim][Br], the ionic liquid monomer [Veim][Br] (1g, 0.005mol) and ethanol (1g, 0.022mol) were purged with Nitrogen gas, N_2 for 30min. AIBN (20mg, 0.12mmol) and TRIM (50mg, 0.15mmol) was added to the solution, purged with N_2 for another 10min and stirred in an oil bath at 65 °C for 30 minutes. After polymerization, the elastic gel particles formed was diluted with the reaction solvent and was poured into acetonitrile to precipitate the Poly[VEIM][Br]. The polymer was washed three times before drying under vacuum at 70°C. The resulting polymer weigh 1.1g (Figure 4.11) was stored in desiccator.

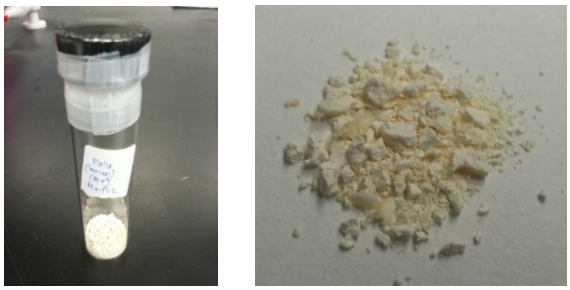


FIGURE 4.11. Poly(1-Vinyl-3-ethylimidazalioum bromide)

On the other hand, Poly(1-Vinyl-3-ethylimidazalioum bromide) incorporating activated carbon, was also synthesized by conventional free-radical polymerization. The polymerization of ionic liquid monomer [Veim]Br incorporating activated carbon was also initiated by addition of 2,2'-azobis(isobutyronitirile) (AIBN) together with Trimethylolpropane tri-metacrylate (TRIM) as the crosslinker. The

synthesis steps are the same as in the synthesis of Poly[Veim][Br] except that in the early step, activated carbon with 5 weight percent (50mg) was added together with ionic liquid monomer [Veim][Br] in the ethanol as the solvent. The resulting polymer weigh 1.2g (Figure 4.12) was stored in desiccator.

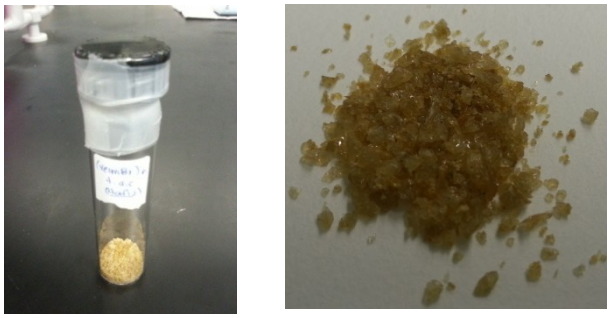


FIGURE 4.12. Poly(1-Vinyl-3-ethylimidazalioum bromide) Incorporating Activated Carbon

The composition of the polymer materials synthesized has been analyzed using energy dispersive X-ray spectroscopy, EDX. The EDX analysis was carried out in order to understand the qualitative elemental composition of the polymer materials.

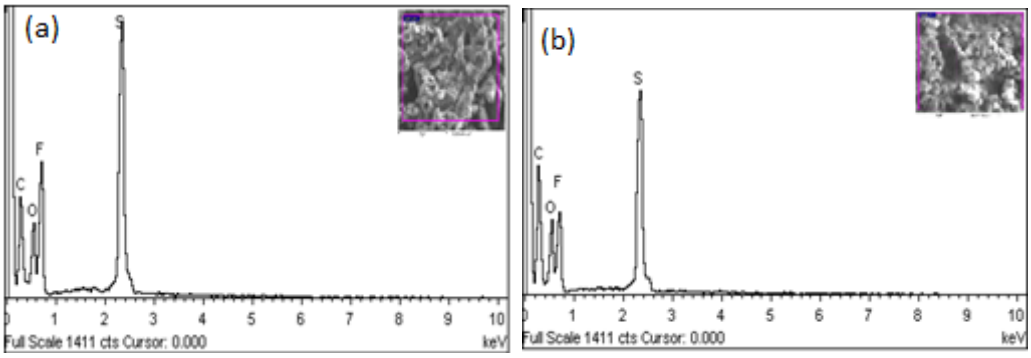


FIGURE 4.13. EDX Analysis of (a) Poly[Veim][Br] (b) Poly[Veim][Br] Incorporating Activated Carbon

From Figure 4.13, the major components exist in both polymer materials are Carbon, Oxygen, Fluorine and Sulfur. It can be observed that the percentage of carbon is higher in the Poly[Veim][Br] incorporating activated carbon which is 49.30% than Poly[Veim][Br] which is 42.46% (Appendix A1&A2). This reveals that activated carbon has actually been successfully incorporated in the Poly[Veim][Br].

In addition, the morphology of the polymer materials synthesized were observed by field emission scanning electron microscope, SEM using acceleration voltage of 2kV and 4kV respectively on the polymer materials surface. The results will analyze the structure of Poly[Veim][Br] (Figure 4.14) with Poly[Veim][Br] incorporating activated carbon (Figure 4.15) at magnification of 200, 1000, 5000 and 10000.

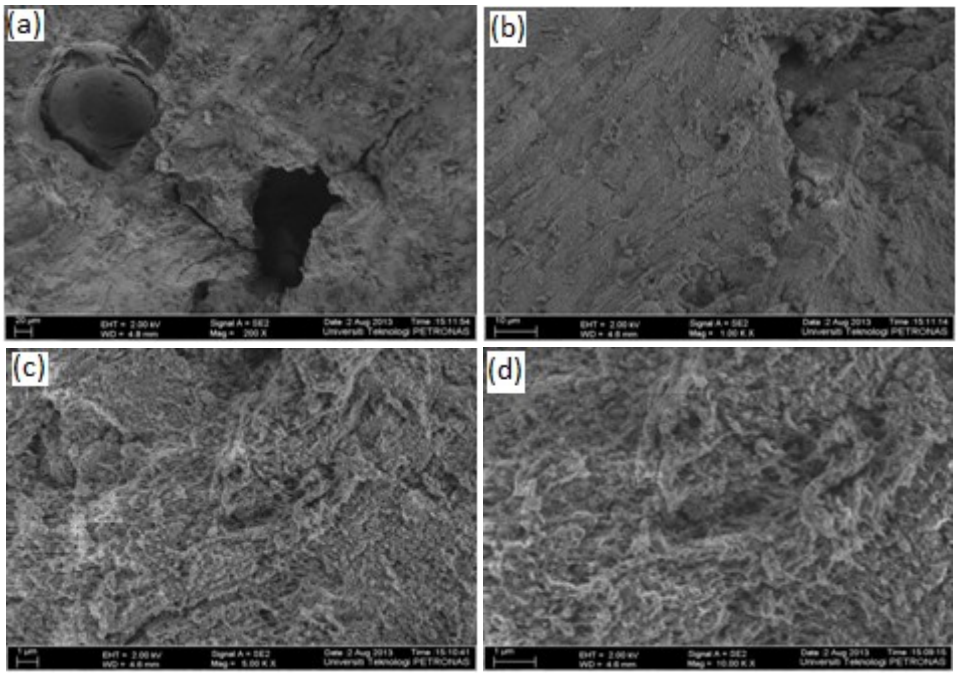


FIGURE 4.14. SEM Images of the Surface of Poly[Veim][Br] at Magnification of (a) 200 (b) 1000 (c) 5000 (d) 10000

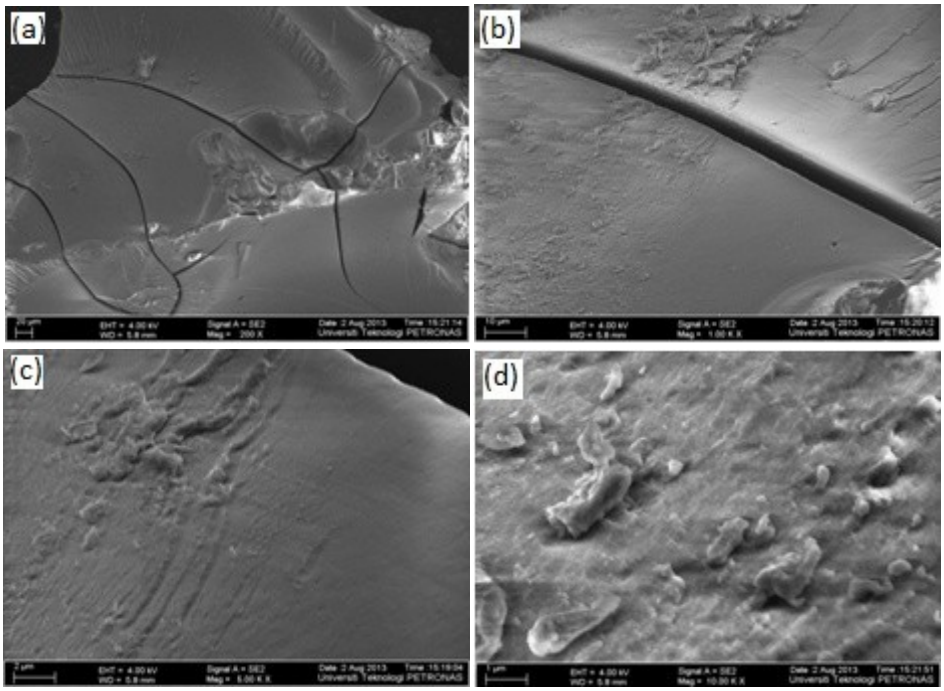


FIGURE 4.15. SEM Images of the Surface of Poly[Veim][Br] Incorporating Activated Carbon at Magnification of (a) 200 (b) 1000 (c) 5000 (d) 10000

From the SEM images in Figure 4.14, it can be observed that the surface of the polymer material Poly[Veim][Br] synthesized is sufficiently porous. At the magnification of 200, there is formation of pore in the polymer molecules. Further magnification at 1000, 5000 and 10000 shows that polymer surface is very porous.

On the other hand, the latter SEM images from Figure 4.15 shows that the surface of the polymer material Poly[Veim][Br] incorporating activated carbon synthesized at magnification of 200 is more rigid, even and dense compared to the Poly[Veim][Br]. The further magnification at 1000, 5000 and 10000 results reveals that by incorporating activated carbon, it leads to the collapse and reduce in the number of large pore demonstrating the role of activated carbon in the structure of the polymer material. Activated carbon incorporated into the polymer material is microporous with large surface area hence it has covered uniformly on the surface of the polymer material and therefore results in less big porous structure.

4.4 SYNTHESIS OF IONIC LIQUID POLYMERS POLY[VEIM][TF_2N] AND POLY[VEIM][TF_2N] INCORPORATING ACTIVATED CARBON

The Poly(1-Vinyl-3-ethylimidazalioum bis(trifluoromethylsulfonylimide)), poly[VEIM][Tf_2N] was synthesized by conventional free-radical polymerization. The polymerization of ionic liquid monomer [Veim][Tf_2N] was initiated by addition of 2,2'-azobis(isobutyronitirile) (AIBN) together with Trimethylolpropane tri-metacrylate (TRIM) as the crosslinker. The synthesis route is illustrated as in Figure 4.16.

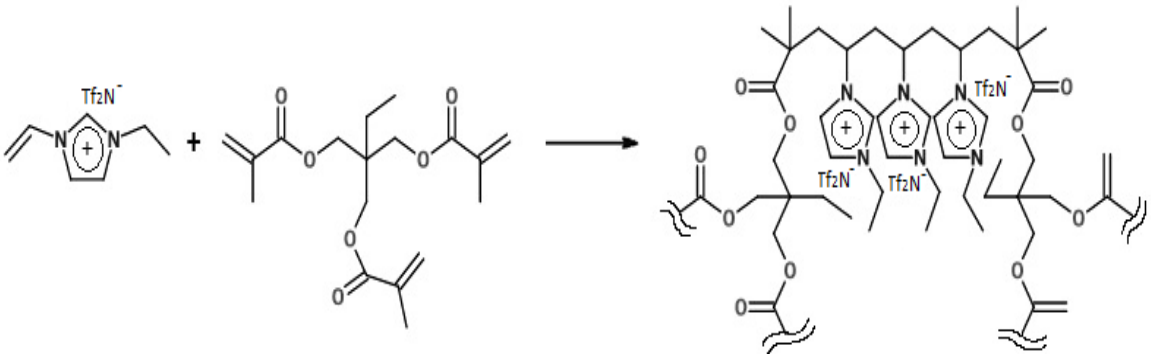


FIGURE 4.16. Synthesis Route of Poly(1-Vinyl-3-ethylimidazalioum bis(trifluoromethylsulfonylimide)

In the synthesis of the Poly[Veim][Tf_2N], the ionic liquid monomer [Veim][Tf_2N] (1g, 0.003mol) and ethanol (1g, 0.022mol) were purged with N_2 for 30min. AIBN (20mg, 0.12mmol) and TRIM (50mg, 0.15mmol) was added to the solution, purged with N_2 for another 10min and stirred in an oil bath at 65°C for 30 minutes. After polymerization, the elastic gel particles formed was diluted with the reaction solvent and was poured into distilled water to precipitate the Poly[VEIM][Tf_2N]. The

polymer was washed three times before drying under vacuum at 70°C. The resulting polymer weigh 1.05g (Figure 4.17) was stored in desiccator.

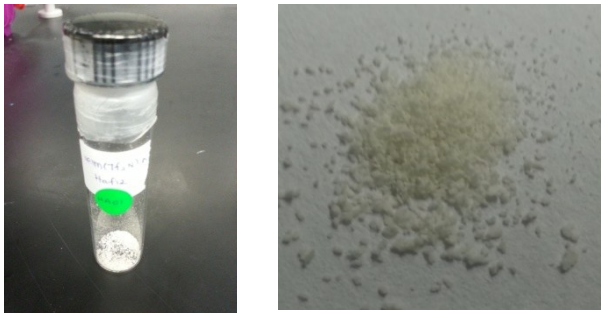


FIGURE 4.17. Poly(1-Vinyl-3-ethylimidazalioum bis(trifluoromethylsulfonylimide)

On the other hand, Poly(1-Vinyl-3-ethylimidazalioum bis(trifluoromethyl-sulfonylimide) incorporating activated carbon, were also synthesized by conventional free-radical polymerization. The polymerization of ionic liquid monomer [Veim][Tf_2N] incorporating activated carbon was also initiated by addition of 2,2'-azobis(isobutyronitirile) (AIBN) together with Trimethylolpropane tri-metacrylate (TRIM) as the crosslinker. The synthesis steps are the same as in the synthesis of Poly[Veim][Tf_2N] except that in the early step, activated carbon was added together with ionic liquid monomer [Veim][Tf_2N] in the ethanol as the solvent. The resulting polymer weigh 1.15g (Figure 4.18) was stored in desiccator.

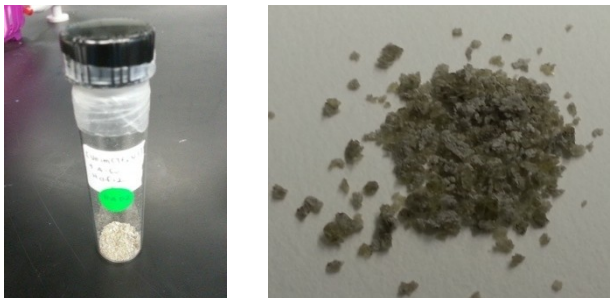


FIGURE 4.18. Poly(1-Vinyl-3-ethylimidazalioum bis(trifluoromethylsulfonylimide) Incorporating Activated Carbon

The composition of the polymer materials synthesized has again been analyzed using energy dispersive X-ray spectroscopy, EDX. The EDX analysis was carried out in order to understand the qualitative elemental composition of the polymer materials.

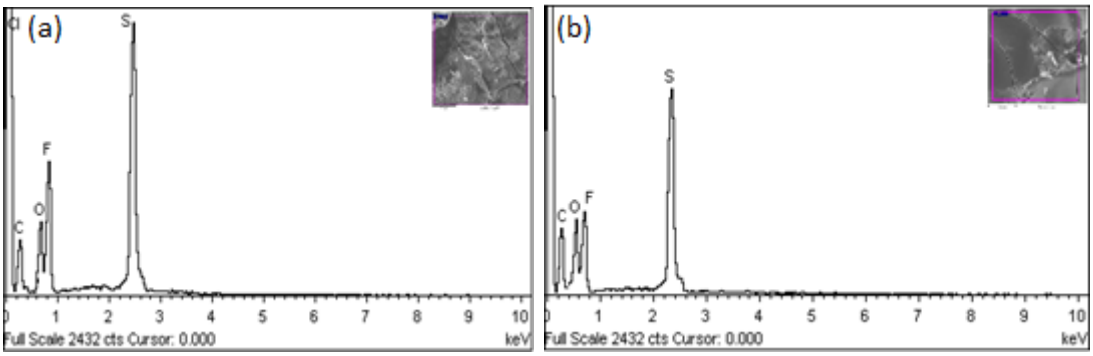


FIGURE 4.19. EDX Analysis of (a) Poly[Veim][Tf_2N] (b) Poly[Veim][Tf_2N] Incorporating Activated Carbon

From Figure 4.19, the major components exists in both polymer materials are Carbon, Oxygen, Fluorine and Sulfur. It can be observed that the percentage of carbon is higher in the Poly[Veim][Tf_2N] incorporating activated carbon which is 50.10% compared to Poly[Veim][Tf_2N] which is 42.54% (Appendix A3&A4). This reveals that activated carbon has actually been successfully incorporated in the Poly[Veim][Tf_2N].

Furthermore, the morphology of the polymer materials synthesized were observed by field emission scanning electron microscope, SEM using acceleration voltage of 5kV on the polymer materials surface. The results will analyze the structure of Poly[Veim][Tf_2N] (Figure 4.20) with Poly[Veim][Tf_2N] incorporating activated carbon (Figure 4.21) at magnification of 200, 1000, 5000 and 10000.

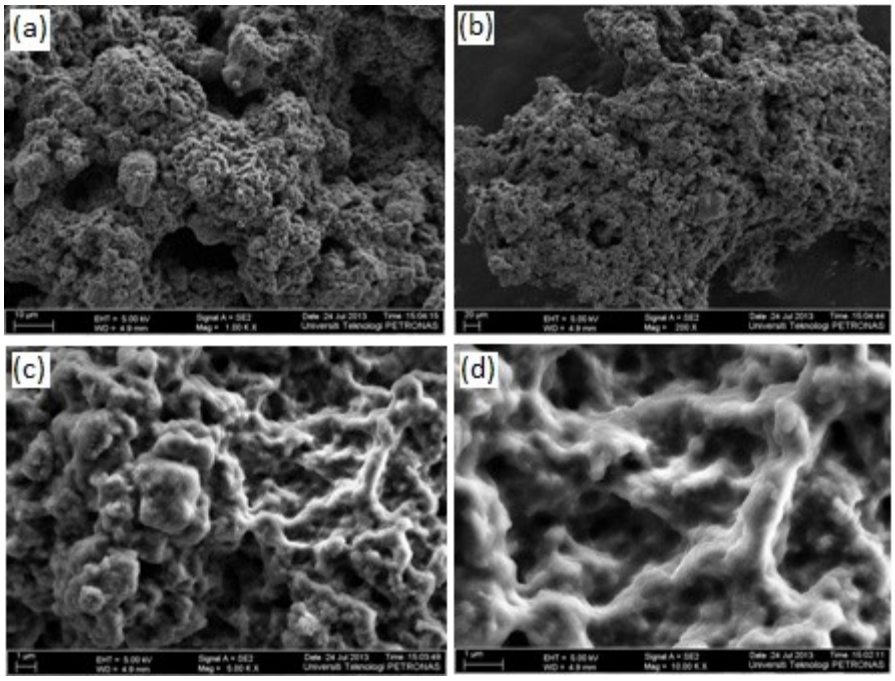


FIGURE 4.20. SEM Images of the Surface of Poly[Veim][Tf_2N] at Magnification of (a) 200 (b) 1000 (c) 5000 (d) 10000

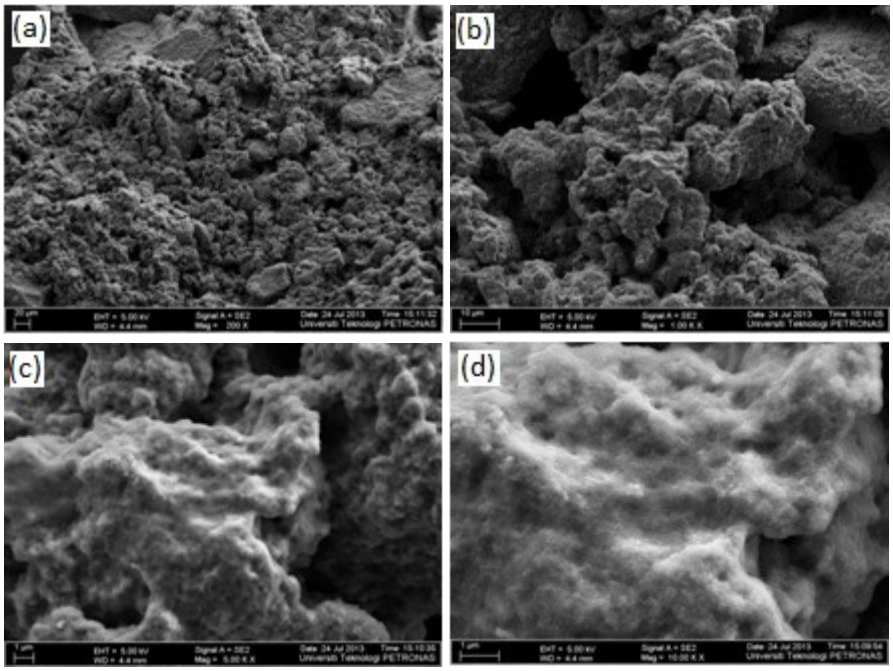


FIGURE 4.21. SEM Images of the Surface of Poly[Veim][Tf_2N] Incorporating Activated Carbon at Magnification of (a) 200 (b) 1000 (c) 5000 (d) 10000

From the SEM images in Figure 4.20, it can be observed that the surface of the polymer material Poly[Veim][Tf_2N] synthesized is very porous as compared to Poly[Veim][Br]. At the magnification of 200, there is numerous formation of large pore in the polymer molecules. Further magnification at 1000, 5000 and 10000 shows that polymer surface is sufficiently very porous.

Moreover, the latter SEM images from Figure 4.21 shows that the surface of the polymer material Poly[Veim][Tf_2N] incorporating activated carbon synthesized has less big pores compared to the Poly[Veim][Tf_2N]. The further magnification at 1000, 5000 and 10000 results reveals that again by incorporating activated carbon, again it leads to reduce in the number of large pore on the polymer surface indicating the role of activated carbon in the structure of the polymer material. Activated carbon has been found to cover uniformly on the surface of the polymer material which has results in less big porous structure as the activated carbon itself has a microporous internal structure.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

In conclusion, the objective of this research project which is to synthesize the ionic liquid polymer incorporating activated carbon has been successfully achieved. The two polymer materials, Poly[Veim][Br] and Poly[Veim][Tf_2N] incorporating activated carbon were synthesized through free radical polymerization with 2,2'-azobis(isobutyronitirile) (AIBN) as the initiator together with trimethylolpropane trimetacrylate (TRIM) as the crosslinker.

The elemental composition of both polymer materials synthesized were examined using energy dispersive X-ray spectroscopy (EDX), showing that the weight and atomic percent of carbon is higher in the Poly[Veim][Br] and Poly[Veim][Tf_2N] incorporating activated carbon compared to Poly[Veim][Br] and Poly[Veim][Tf_2N] respectively. This proves that activated carbon has been successfully incorporated into both of the polymer molecules. Furthermore, the morphology of both polymer materials has been investigated using field scanning emission scanning electron microscopy, (SEM). SEM images at 10000 magnification have reveal that by incorporating activated carbon, it leads to the collapse and reduce in the number of large pore demonstrating the role of activated carbon in the structure of the polymer material by uniformly covered on both of the polymer surfaces.

In future work, evaluating the solubility of the CO_2 in the polymer materials synthesized is important. This is to understand the performance of the polymer materials for CO_2 capture and separation in order to be applied in the industry. Developing the polymer material with low cost and low viscosity is also vital as it will increase its efficiency and decrease the energy demand for desorption of the polymer material after the CO_2 separation as CO_2 desorption process is also crucial with respect to the energy consumption required by industries.

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APPENDICES

Appendix A: Energy Dispersive X-Ray (EDX) Analysis of Polymer Materials Synthesized

A1: Elemental Composition of Poly[Veim][Br]

Elements	Weight Percent (%)	Atomic Percent (%)
Carbon	42.46	54.48
Oxygen	15.95	15.36
Fluorine	30.78	24.97
Sulfur	10.80	5.19

A2: Elemental Composition of Poly[Veim][Br] Incorporating Activated Carbon

Elements	Weight Percent (%)	Atomic Percent (%)
Carbon	49.30	60.38
Oxygen	18.96	17.53
Fluorine	23.10	17.98
Sulfur	8.91	4.11

A3: Elemental Composition of Poly[Veim][Tf_2N]

Elements	Weight Percent (%)	Atomic Percent (%)
Carbon	42.54	54.40
Oxygen	16.05	15.11
Fluorine	31.86	25.64
Sulfur	9.55	4.85

A4: Elemental Composition of Poly[Veim][Tf_2N] Incorporating Activated Carbon

Elements	Weight Percent (%)	Atomic Percent (%)
Carbon	50.10	60.61
Oxygen	17.17	17.15
Fluorine	25.10	18.26
Sulfur	7.63	3.98